

## Certificate of Analysis

## Standard Reference Material® 1660a

## Methane and Propane in Air

(Nominal Amount-of-Substance Fraction: Methane – 4 μmol/mol; Propane – 1 μmol/mol)

This certificate reports the certified values for designated cylinders of Lot 13-XX-E.

This Standard Reference Material (SRM) is a primary gas mixture that, the amount-of-substance fraction expressed as concentration [1], may be related to secondary working standards. This SRM is intended for the calibration of instruments used for methane/propane determinations and for other applications.

This SRM mixture is supplied in a DOT 3AL specification aluminum (6061 alloy) cylinder with a water volume of 6 L. Mixtures are shipped with a nominal pressure exceeding 12.4 MPa (1800 psi), which provides the user with 0.73 m³ (25.8 ft³) of useable mixture. The cylinder is the property of the purchaser and is equipped with a CGA-590 brass valve, which is the recommended outlet for this methane/propane mixture. NIST recommends that this cylinder **NOT** be used below 0.7 MPa (100 psi).

**Certified Value:** This SRM mixture has been certified for methane and propane concentrations. The certified values, given below, apply to the identified cylinder and NIST sample number.

Methane Concentration: 3.94  $\mu$ mol/mol  $\pm$  0.02  $\mu$ mol/mol Propane Concentration: 0.972  $\mu$ mol/mol  $\pm$  0.005  $\mu$ mol/mol

Cylinder Number: NIST Sample Number:

The uncertainty of each certified value includes the estimated uncertainties in the NIST standards, the analytical comparisons to the lot standard (LS), and the uncertainty of comparing the LS with each of the mixtures comprising this lot. This uncertainty is expressed as an expanded uncertainty  $U = ku_c$ , with  $u_c$  determined by experiment and a coverage factor k = 2. The true values for the methane and propane amount-of-substance fractions are asserted to lie in the interval defined by the certified value  $\pm U$  with a level of confidence of approximately 95 % [2].

**Expiration of Certification:** This certification is valid until **01 January 2010**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification will be nullified if the SRM is contaminated or modified.

**Hydrotest Date:** 03/94 **Blend Date:** 06/94

**Cylinder and Gas Handling Information:** NIST recommends the use of a high purity, two-stage pressure regulator with a stainless steel diaphragm and CGA-590 outlet to safely reduce the pressure and to deliver this SRM mixture to the instrument. The regulator should be purged several times to prevent accidental contamination of the sample.

The analytical measurements leading to the certification of this current SRM lot were performed by G.C. Rhoderick of the NIST Analytical Chemistry Division.

The overall direction and coordination of the technical work for required for certification of this SRM were performed by F.R. Guenther of the NIST Analytical Chemistry Division.

Stephen A. Wise, Chief Analytical Chemistry Division

Robert L. Watters, Jr., Chief Measurement Services Division

Gaithersburg, MD 20899 Certificate Issue Date: 24 June 2005 See Certificate Revision History on Last Page

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The support aspects involved in the issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by C.S. Davis of the NIST Measurement Services Division.

**Mixture Preparation:** The gas mixtures comprising this SRM lot were prepared in accordance with NIST technical specifications by a commercial specialty gas vendor under contract to NIST. The specifications stipulate that each SRM mixture be identical in methane/propane concentration and stable with time.

**Analytical Methods:** Analyses of the methane/propane concentration for this lot of cylinders were conducted by inter-comparing each cylinder mixture to a representative cylinder chosen from the lot, the lot standard (LS), using a research gas chromatograph (GC) equipped with a flame ionization detector (FID). Assignment of the methane and propane concentrations to the LS was accomplished by inter-comparison to primary gravimetric standards using GC/FID.

**Homogeneity Analysis:** Each of the methane/propane mixtures that comprise this SRM lot was compared to the LS using GC/FID. An analysis of variance indicated that sample-to-sample methane/propane concentration differences were statistically insignificant. This indicates that within the precision of the NIST measurements, all of the cylinders comprising this SRM lot have identical methane/propane concentrations. Therefore, one concentration has been assigned to the entire SRM lot.

**Methane/propane Concentration Value Assignment:** The certified methane and propane concentrations for this SRM lot were computed from the assigned concentration for the LS and the homogeneity analysis.

**Stability:** Periodic analyses of SRM units from this lot are performed at NIST to monitor stability. If significant changes in the methane or propane concentrations are observed, the purchaser will be notified. Refer to the "Cylinder and Gas Handling Information" section for proper handling of this SRM.

**Other Analyses:** Additional analyses performed during the certification procedure are given below. The concentrations reported below are **NOT** certified values. These values are given for informational purposes only.

| Constituent              | Concentration    | Method            |
|--------------------------|------------------|-------------------|
| Argon                    | 0.92 % mol/mol   | Mass Spectrometry |
| Carbon Dioxide           | 350 μmol/mol     | Mass Spectrometry |
| Oxygen                   | 20.9 % mol/mol   | Mass Spectrometry |
| Total Other Hydrocarbons | < 0.010 µmol/mol | GC/FID            |
| (expressed as methane)   |                  |                   |

## REFERENCES

- [1] Taylor, B.N.; Guide for the Use of the International System of Units (SI); NIST Special Publication 811, 1995 ed. (1995).
- [2] ISO; Guide to the Expression of Uncertainty in Measurement; ISBN 92-67-10188-9, 1st ed., International Organization for Standardization: Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results; NIST Technical Note 1297, U.S. Government Printing Office: Washington, DC (1994); available at <a href="http://physics.nist.gov/Pubs/">http://physics.nist.gov/Pubs/</a>.

**Certificate Revision History:** 24 June 2005 (This revision reflects an extension in the certification period for cylinders listed in this document); 16 July 2001 (Original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at <a href="http://www.nist.gov/srm">http://www.nist.gov/srm</a>.

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